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DEPARTMENT OF COMMERCE
BUREAU OF STANDARDS
WASHINGTON

Letter
Circular
LC 300

June 15, 1926.

Measurement of Crankcase Dilution. Capillary Funnel Method.

This approximate method has been developed to afford an easy rapid means of measuring the percentage of fuel diluent in used crankcase oils. Results are usually within 1% of the truth and seldom so far as 2% out.

The method has been in use in the automotive laboratory of the Bureau of Standards for about two years and satisfactory apparatus for the test is now commercially available. There seems to be a need for a suitable simple method of measuring dilution and this description has been prepared to meet the many requests which have been received for information regarding this method.

Apparatus

1 - 250 cc distillation flask

1 - Capillary funnel

1 - 350 watt embedded type electric heater or suitable gas burner.

1 - Condenser

1 - 100 cc graduated cylinder

5 grams No. 3 steel wool

1 Cork to fit flask

1 Wire hook to handle steel wool

1 Wire probe No. 24 or 26 B&S gage to pass through capillary.

Flask

This is the 250 cc Saybolt distilling flask as specified for A.S.T.M. Method D158-25T with about 6 cm of end of vapor tube turned down to meet vertical condenser, as shown in blueprint in which principal dimensions are also indicated.

Funnel

This is a stock 60° funnel about 4 cm across top with a stem from 4 to 8 cm long, to which is sealed a 6 cm length of capillary tubing having a bore of 0.06 cm ±0.01 cm. A small hole 1 to 1.5 mm diameter, drilled about 1 cm above top of stem (see figure) to take care of flooding with oils showing high dilution is convenient but not essential.

Heater

This is of the embedded Bureau of Mines* type formed to fit the flask bottom as shown in the figure and having a capacity of about 350 watts. Apparently distributed heat is desirable as a bare wire heater run at high temperatures gives high results due to cracking of the oil.

Condenser

A metal condenser with central tube about 1 cm (3/8 in.) or less in diameter and having at least 15 cm (6 in.) jacketed with water at a temperature below 30°C (86°F), or a glass condenser of 20 cm (8 in.) jacketed length, gives adequate condensation. If the condenser jacket has a capacity of 75 cm^3 or more, circulation of water is not necessary except for continuous use or where the sample contains considerable water. A straight condenser set at an angle of 75° with the vertical may be used with a flask having a straight vapor tube if desired.

Receiver

This is an ordinary 100 cc graduate.

Steel Wool

This is No. 3 grade, coarse, formed into a cylinder which fits snugly into the flask neck and extends from outlet of vapor tube almost to the bottom of the flask.

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*Bull. Bureau of Mines No. 207.

Operation

Electric Heating

Connect heater to 110 volt mains (or 220 volt if heater is designed for that voltage). Measure 100 cc of the oil to be tested into the flask and place steel wool in neck with top just below vapor outlet. Cork flask tightly and when heater is hot (about 5 minutes) place flask on heater with vapor tube entering condenser, no seal is necessary at this point, and place graduate and funnel as shown. Distillation will begin in from one to three minutes. If much diluent is present the funnel will fill rapidly and overflow into graduate through the small hole. If the funnel does not have this hole it should be moved to one side until the excessive flow of distillate begins to slacken, and then replaced.

As the distillation proceeds the level of liquid in the funnel stem will drop until it is very near the top of the capillary or it may drop below this point and entrain air bubbles in the capillary. Finally as the oil begins to distill over, the head of liquid on the stem will begin to rise rapidly due to the increased viscosity of the oil and the increased rate of distillation.

The volume, number of cubic centimeters, of distillate in the receiver, corrected for the water which may have distilled, at the time the rapid rise of head takes place, is taken as the percentage of diluent in the original charge. Usually during this period the head increases 2 or 3 cm by the time one cubic centimeter has run into the graduate so that the end point is sufficiently definite.

The flask may now be taken from the heater and when partially cooled the steel wool removed with a small wire hook, set aside to drain for the next test, and the warm oil allowed to drain from the flask.

Gas Heating

In this case the procedure is as above except that the gas burner is adjusted to cover the lower 1/3 or 1/4 of the flask with a slightly yellowish flame. A "hard" flame with a well defined cone is undesirable and a shield should be used to guard against ignition of the distillate.

After the period of flooding is over the rate of heating should be so adjusted as to maintain a head of about 1.5 cm (1/2 in.) on the capillary. Towards the end of the test when this head tends to drop the heat should be increased gradually to oppose this action. With high boiling oils the drop

will probably persist until air bubbles are trapped. The rate of heat supply should be such that the rapid rise occurs in not more than 3 minutes from the time entrainment of bubbles starts. Too little heat will permit excessive cracking of the oil and high apparent dilution results will ensue. A setting of the gas flame which will evaporate a charge of water from the flask at a rate of about 5 cc per minute is usually satisfactory. Test requires about 15 minutes.

Accuracy

The capillary funnel method may be expected to give results certainly within 2% of the truth with the oils ordinarily encountered in automobile crankcase use. Usually results are within 1%; that is, an indicated dilution of 15% is likely to be within the limits 14-16%. Reproducibility of results with a particular sample are within 1%.

Special Notes

Water

Water is often present in samples of used crankcase oil and interferes to some extent with the distillation. The presence of water in the oil is evidenced during distillation by sharp popping sounds and miniature explosions in the oil. If much water is present the contents of the flask may froth up and, if not prevented, pass as froth through the vapor tube to the condenser. Sometimes also droplets of water condensing on the cooler parts of the flask will drop back into the hot oil and vaporize with considerable violence.

Ordinarily appreciable amounts of water will not be found in summer samples or in samples of oil in which the dilution does not exceed 10 to 12%. If it is not desired to evaluate water the sample may be allowed to settle overnight after which a practically water free portion may usually be poured off the top for distillation.

If wet samples must be distilled or if water is shown by expressive frothing during distillation the addition of two or three drops of castor oil to the charge will tend to suppress foaming and permit the distillation of quite large percentages of water. In exceptional cases it may be necessary to reduce the rate of distillation temporarily by switching off the heat, raising the flask a short distance (1/4 to 1/2 inch) from the heater, or reducing the gas supply until the water has been distilled off. This is indicated by the disappearance of foam from the surface of the oil in the flask and a hissing or crackling sound in the vapor tube and condenser due to the contact of hot vapor with condensed water.

Water often carries over traces of sludge which tend to clog the capillary consequently the small wire probe should be used to keep the bore clear especially when wet oils are being distilled.

Cleaning Flasks

It is not necessary to clean the flasks after each test as draining when hot is sufficient since the residual oil contains no diluent. If the flask is rinsed out with gasoline, however, it must be thoroughly dried before use. The black deposits which are built up in the flasks by continued use may be removed by filling the flask with alcoholic caustic soda solution, allowing to stand over night (heating the solution will accelerate the action) and rinsing out with water and coarse sand. This cleaning solution may be made up by taking a water solution of sodium hydroxide (ordinary lye as sold in tin cans for household use is equally effective) containing about 50% by weight of the hydroxide and diluting this to about four times its volume with pure or denatured ethyl alcohol.

The cleaning solution may be re-used almost indefinitely but should be kept stoppered to prevent loss by evaporation.

Theory of the Method

The diluent in crankcase oils ordinarily consists of constituents from the last half of the fuel distillation range. The bulk of this material approximates in composition that coming off at about the 85-90% point of the Engler distillation.

This material has lower boiling points than even the light fractions of the oil and is decidedly less viscous. Consequently when a diluted oil is distilled the first portions coming over will be largely diluent and will be of low viscosity but a pronounced increase in viscosity will occur when the oil begins to come over.

In the distillation it is, however, desirable to get some fractionation of the vapor to effect better separation of oil and diluent. This is promoted by a moderate rate of distillation and by the use of the steel wool which also helps to prevent dropping back of water with very wet samples.

The head of liquid on the capillary is proportional to the rate of flow of distillate from the condenser, to the viscosity of the distillate and to the resistance which the capillary offers to the passage of distillate. This resistance of the capillary is proportional directly to its length and inversely to the fourth power of the diameter of the bore.

Consequently bore size must be kept within reasonable limits.

In the test as specified the rate of supply of heat in the electrical method, and the rate of distillation with gas heating are kept approximately constant so that for a given capillary the change in viscosity due to the presence of considerable amounts of oil in the distillate will show up as an increase in head of distillate on the capillary.

Petroleum lubricating oils will crack or decompose at high temperatures giving a low viscosity product and a carbonaceous residue. Unfortunately with the oils of higher boiling points the temperatures necessary to distill off all of the diluent fall within the lower range of cracking temperatures. The heavy steam like vapor issuing from the condenser near the end of the test is evidence of cracking.

The amount of cracking produced is a function of both the temperature and the time so that a very slow rate of heating in this region leads to more cracking than a faster rate and for this reason insufficient heat supply at this time will lead to excessively high apparent dilution. (See section on "Gas Heating"). On the other hand, a too fast rate of heating will lead to error due to insufficient fractionation of the vapor. The specifications for the method, rate of heat supply, size of capillary, etc., have been chosen to give optimum results over the ordinary range of oils in common use. In the electrical method the requirements as to rate of heating are taken care of by the capacity and construction of the heater. Consequently this is the preferable method, especially in the hands of unskilled operators. Concentrated heat as from a sharply defined gas cone or an electric heater where there is direct radiation from very hot wires seems to produce high local heating in the film of oil adhering to the flask wall and so lead to cracking and to erroneous, high, results.

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Note: At this time apparatus as indicated below has been found satisfactory for this test.

Flask and Capillary Funnel -
Corning Glass Works,
501 Fifth Avenue,
New York City, N. Y.

Complete apparatus electric heating -
American Instrument Company,
1220 D Street, N. W.,
Washington, D. C.

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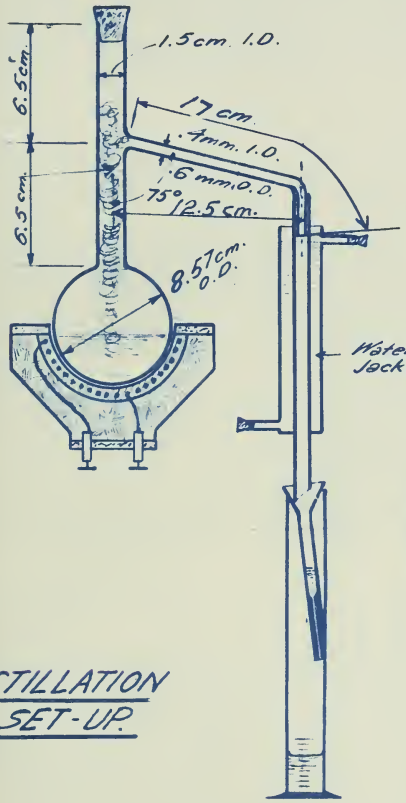
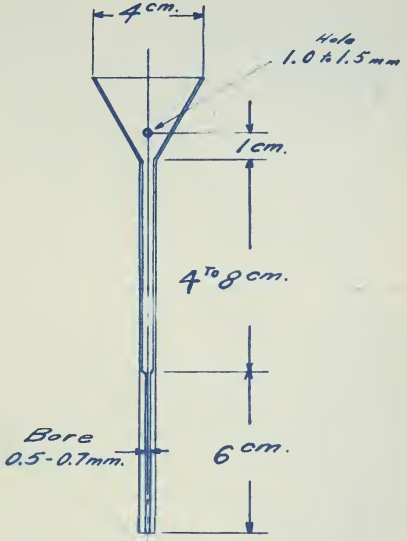
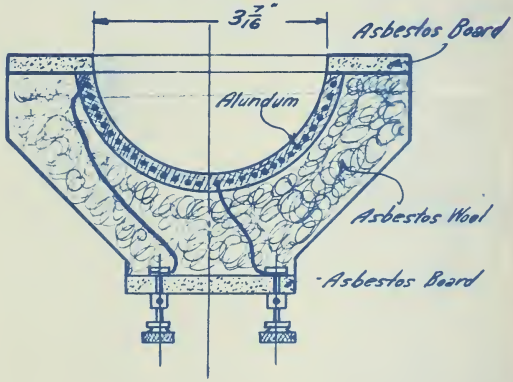


DISTILLATION SET-UP.



CAPILLARY FUNNEL



ELECTRIC HEATER

110 V - 350 WATTS

CRANKCASE DILUTION (CAPILLARY FUNNEL METHOD)